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(11)Publication number:

2002-167257

(43)Date of publication of application: 11.06.2002

(51)Int.CI.

CO4B 24/26 CO4B 24/02 CO8F220/28 CO8F290/06 // CO4B103:40

(21)Application number: 2001-284445

(22)Date of filing:

19.09.2001

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(30)Priority

Priority number : 2000283349

Priority date: 19.09.2000

Priority country: JP

(54) CEMENT DISPERSANT

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a cement dispersant preventing slump loss at high temperature while keeping high fluidity at filling.

SOLUTION: The cement dispersant obtained by copolymerizing the specific polymer (A1) such as an ethylenic unsaturated carboxylic acid derivative having polyoxyalkylene group and the specific polymer (A2) such as (meth)acrylic acid, contains at least one compound selected from a group consisting of a copolymer mixture, the mole ratio (A1)/(A2) of which is changed at least one time in the middle of reaction, a salt of the copolymer mixture obtained by neutralizing by alkali, oxycarboxylic acid or its salt, saccharides and suger- alcohol.

LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

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CLAIMS

[Claim(s)]

[Claim 1] Carry out copolymerization of at least one sort (A2) of a monomer expressed with at least one sort (A1) of a monomer expressed with the following general formula (a1), and the following general formula (a2), and it is obtained. And below copolymer mixture salt [that neutralized with alkali the copolymer mixture or this copolymer mixture from which said monomer (A1) and mole-ratio (A1)/(A2) of (A2) are changing in the middle of the reaction once [at least] The cement dispersing agent containing the compound (B) more than a kind chosen from the group which becomes] called copolymer mixture (A) in both from hydroxy acid or its salt, a saccharide, and sugar-alcohol.

[Formula 1]

(The inside of a formula, the oxy-alkylene group of the number AO:carbon numbers 2-4 of R1, an R2:hydrogen atom, the number R3:hydrogen atom of methyl-group m:0-2, -COO(AO) nXp:0, or 1, or several X of oxy-styrene radical n:2-300: Express a hydrogen atom or the alkyl group of carbon numbers 1-18.)

(The inside of a formula, R4-R6: It is a hydrogen atom, a methyl group, or (CH2) m1COOM2, and m (CH2)1COOM2 may form COOM1 or other m(CH2)1COOM(s)2, and an anhydride, and M1 and M2 of those radicals do not exist in that case.)

M1, M2: Express a hydrogen atom, alkali metal, alkaline earth metal, ammonium, an alkylammonium radical, or the number of permutation alkylammonium radical m1:0-2.

[Claim 2] The cement dispersing agent according to claim 1 which has the average weight ratio (XI) of a monomer (A2) to all the monomers for manufacturing copolymer mixture (A) in the range of 1-30 (% of the weight).

[Claim 3] The cement dispersing agent according to claim 1 or 2 whose hydroxy acid is more than a kind chosen from a gluconic acid, glucoheptonic acid, an arabonic acid, a malic acid, and a citric acid. [Claim 4] The cement dispersing agent of claim 1-3 whose saccharide is a compound more than a kind chosen from monosaccharide, oligosaccharide, and polysaccharide given in any 1 term.

[Claim 5] The cement dispersing agent of claim 1-4 whose sugar-alcohol is a sorbitol given in any 1 term.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to a cement dispersing agent. [0002]

[Description of the Prior Art] As a cement dispersing agent, the copolymer (henceforth an acrylic-acid system) manufactured from the polyalkylene glycol monochrome (meta) acrylic ester system monomer and the acrylic-acid (meta) system monomer is known. In this kind of copolymer, it is changing the number of addition mols (it expressing with n hereafter) and monomer ratio of alkylene oxide (it is called Following AO), and it is indicated that it is possible to give the characteristic engine performance (JP,58-74552,A, JP,8-12396,A, etc.). Such a polycarboxylic acid system cement dispersing agent of AO addition mold is excellent in cement dispersibility, the fluidity of the fresh condition of a cement constituent, segregation resistance, and hardening on-the-strength manifestation nature compared with the cement dispersing agent of the naphthalene system used from the former, or a melamine system. Furthermore, in this industry, about this AO addition type of polycarboxylic acid system cement dispersing agent, examination which raises those engine performance, such as high pump pumpability and versatility corresponding to diversification of manufacture conditions, is advanced, and we found out that these engine performance improved by adjusting a copolymerization ratio (Japanese Patent Application No. No. 361108 [11 to]).

[0003]

[Problem(s) to be Solved by the Invention] However, the further improvement in the fluid fall (slump loss) prevention ability with the passage of time under the elevated temperature to which concrete temperature exceeds 30-degree-C order is desired. Especially, in a system with required maintaining a high fluidity also at the time of restoration, the slump loss under an elevated temperature poses a very big problem like the remarkable hi-performance concrete of spread through common freshly mixed concrete, freshly mixed concrete, and precast concrete.

[0004] It is offering the cement dispersing agent which can prevent the slump loss under an elevated temperature, the technical problem of this invention having the versatility over diversification of manufacture conditions etc., and maintaining the high fluidity at the time of restoration.

[Means for Solving the Problem] This invention carries out copolymerization of at least one sort (A2) of a monomer expressed with at least one sort (A1) of a monomer expressed with the following general formula (a1), and the following general formula (a2), and is obtained. And below copolymer mixture salt [that neutralized with alkali the copolymer mixture (A) or this copolymer mixture from which said monomer (A1) and mole-ratio (A1)/(A2) of (A2) are changing in the middle of the reaction once [at least] the compound more than a kind chosen from the group which becomes] called copolymer mixture (A) in both from hydroxy acid or its salt, a saccharide, and sugar-alcohol -- it is related with (B [it is hereafter called the (B) component]). [0006]

[Formula 3]
$$\begin{array}{ccc}
R^1 & R^2 \\
C = C & (a1)
\end{array}$$

$$\begin{array}{cccc}
R^3 & (CH_2)_m(CO)_2O(AO)_0 - X
\end{array}$$

[0007] (The inside of a formula, the oxy-alkylene group of the number AO:carbon numbers 2-4 of R1, an R2:hydrogen atom, the number R3:hydrogen atom of methyl-group m:0-2, -COO(AO) nXp:0, or 1, or several X of oxy-styrene radical n:2-300: Express a hydrogen atom or the alkyl group of carbon numbers 1-18.)

[0009] (The inside of a formula, R4-R6: It is a hydrogen atom, a methyl group, or (CH2) m1COOM2, and m(CH2)1COOM2 may form COOM1 or other m(CH2)1COOM(s)2, and an anhydride, and M1 and M2 of those radicals do not exist in that case.)

M1, M2: Express a hydrogen atom, alkali metal, alkaline earth metal, ammonium, an alkylammonium radical, or the number of permutation alkylammonium radical m1:0-2. [0010]

[Embodiment of the Invention] [Copolymer mixture (A)] as a monomer (A1) expressed with the general formula (a1) used for manufacture of copolymer mixture (A) A methoxy polyethylene glycol, a methoxy polypropylene glycol, A methoxy polybutylene glycol, a methoxy polystyrene glycol, A piece end alkyl blockade polyalkylene glycol and acrylic acids (meta), such as an ethoxy polyethylene polypropylene glycol, An esterification (half) object with a maleic acid, and a etherification object with allyl alcohol (meta), An acrylic acid, a maleic acid, the ethylene oxide to allyl alcohol (meta), and a propylene oxide addition product are used preferably, R3 has a desirable hydrogen atom, and, as for m, 0 is [p/1] and (meta) desirable. It is the esterification object of alkoxy ** especially a methoxy polyethylene glycol, and an acrylic acid (meta) more preferably.

[0011] AO addition mol several n of the monomer (A1) expressed with a general formula (a1), when it becomes small, it is in the inclination for a cure rate, dispersibility, and viscosity to be reduced, and if n becomes large, these will tend to increase. Therefore, what is necessary is just to choose n in all as the target engine performance.

[0012] For example, when thinking the early-age-strength manifestation nature of concrete as important, it is desirable that it is 80<=n and they are [100<=n and] 110<=n most preferably preferably [it is more desirable and] to 90<=n and a pan. moreover -- since dispersibility falls and the polymerization nature at the time of being manufacture also falls in 300<n -- more -- desirable -- n<=200 -- further -- desirable -- n<=150 -- it is n<=130 especially preferably.

[0013] the case where viscous reduction of concrete is thought as important -- 2<=n<=100 -- desirable -- more -- desirable -- 5<=n<=50 -- it is 5<=n<=30 most preferably. [0014] When it is required to have early-age-strength manifestation nature and viscous reduction, it is desirable to copolymerize the big thing of n and a small thing, and it is desirable especially as a monomer (A1) to use together the monomer (A1-2) expressed with the monomer (A1-1) and the following general formula (a1-2) which are expressed with the following general formula (a1-1). [0015]

[Formula 5]

[0016] (the inside of a formula, a Ra1:hydrogen atom or the oxy-alkylene group of the methyl group AO:carbon numbers 2-4, or an oxy-styrene radical -- desirable -- several X [of carbon numbers 2-3] of oxy-alkylene group n1:12-300 -- 1:hydrogen atom or the alkyl group of carbon numbers 1-18 -- a hydrogen atom or the alkyl group of carbon numbers 1-3 is expressed preferably.)
[0017]

[0018] A hydrogen atom or a methyl group AO: Ra2: the inside of a formula, the oxy-alkylene group or oxy-styrene radical of carbon numbers 2-4, preferably oxy-alkylene group [of carbon numbers 2-3] n the number of 2:2-290 (however, the relation of n1 in a general formula (a1-1) -- n1>n2 and (n1-n2) >=10 -- desirable -- >=30 -- it is >=50 still more preferably.)

X2: a hydrogen atom or the alkyl group of carbon numbers 1-18 -- express a hydrogen atom or the alkyl group of carbon numbers 1-3 preferably.

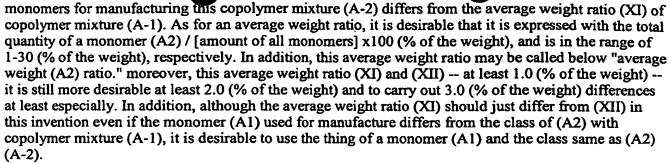
[0019] in this case, both average weight ratio -- desirable -- /(A1-1) (A1-2) =0.1-8 -- it is 0.2-2.5, and being in the range of 0.4-2 preferably especially more preferably. In addition, this average weight ratio is the average of the weight ratio of all the monomers used for a reaction.

[0020] moreover, reaction mole-ratio [of a monomer (A1-1), (A1-2), and (A2)] [(A1-1) + (A1-2)]/(A2) -- desirable -- this mole ratio before and behind change -- at least -- or [any] -- 0.02-4 -- it is 0.05-2.5, and being in the range of 0.1-2 preferably especially still more preferably. This mole ratio before and behind change is being in these range most preferably [both].

[0021] Under such conditions, it is desirable that it is $12 \le n1 \le 300$, $2 \le n2 \le 290$, and $n2+10 \le n1$, and if it is $n2+50 \le n1$ preferably [it is more desirable and] to $n2+30 \le n1$ and a pan, both engine performance will be notably discovered. further -- desirable -- $80 \le n -- 1 \le 300$ and $2 \le n2 \le 30 --$ more -- desirable -- $100 \le n --$ it is choosing $1 \le 300$ and $2 \le n2 \le 10$ to n1 and n2.

[0022] moreover, as a monomer (A2) expressed with the general formula (a2) used for manufacture of copolymer mixture (A) Monocarboxylic acid system monomers, such as an acrylic acid and a crotonic acid, a maleic acid, (Meta) Dicarboxylic acid system monomers, such as an itaconic acid and a fumaric acid, these anhydrides, or a salt, For example, the monochrome by which an alkali-metal salt, an alkaline-earth-metal salt, ammonium salt, and a hydroxyl group may be permuted, JI and trialkyl (carbon numbers 2-8) ammonium salt -- desirable -- more -- desirable -- an acrylic acid (meta), a maleic acid, and a maleic anhydride -- they are acrylic acids (meta) or these alkali-metal salts still more preferably.

[0023] Although copolymer mixture (A) makes the above-mentioned monomer (A1) react preferably by the mole ratio of the range of /(A1) (A2) =0.02-4 and is obtained, these mole-ratio (A1)/(A2) is changing in the middle of the reaction once [at least] (A2). And it is desirable to use together the copolymer mixture (A-2) obtained by average weight ratio (XII) which is different from the average weight ratio (XI) of a monomer (A2) to all the monomers for manufacturing copolymer mixture (A-1) among copolymer mixture (A) in this invention. Copolymer mixture (A-2) namely, the above-mentioned monomer (A1) (A2) It is the copolymer mixture which was made to react preferably by the mole ratio of the range of /(A1) (A2) =0.02-4, and was obtained. These mole-ratio (A1)/(A2) is changing in the middle of the reaction once [at least]. The average weight ratio (XII) of a monomer (A2) to all the



[0024] It is desirable that the average weight ratio (XI) of copolymer mixture (A-1) is 8 - 16 % of the weight further seven to 20% of the weight one to 30% of the weight especially in this invention. And if a combination system is assembled by using this copolymer mixture (A-1) as base resin, the good concrete water reducing agent of the balance of each engine performance can be obtained.

[0025] In this invention, two or more copolymers obtained from two or more monomer mixture with which average weight (A2) ratios differ as copolymer mixture (A-2), respectively can be used. It is desirable to use 1-3 copolymer mixture obtained from 1-3 monomer mixture with which average weight (A2) ratios differ, respectively from a practical field. When using one copolymer mixture as copolymer mixture (A-2) (i.e., when using two copolymer mixture by all), and they are set to copolymer mixture (Ai) and (Aii) for convenience and these (A2) average weight ratios are set to (Xi) and (Xii), respectively, it is 5 <=(Xi) <8 (% of the weight).

It is desirable that it is 8 <=(Xii) <=16. Moreover, when using two copolymer mixture as copolymer mixture (A-2) (i.e., when using three copolymer mixture by all), and they are set to copolymer mixture (Ai), (Aii), and (Aiii) for convenience and these (A2) average weight ratios are set to (Xi), (Xii), and (Xiii), respectively, it is 5 <=(Xi) <8 (% of the weight).

 $8 \le (Xii) \le 16$ (% of the weight)

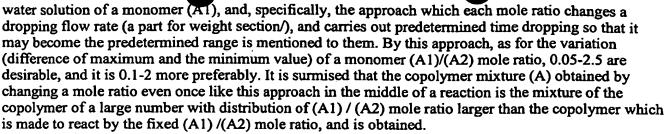
16<(Xiii) <=30 (% of the weight)

It comes out and a certain thing is desirable.

[0026] (A2) Good dispersibility and distributed holdout are discovered at W/C and concrete temperature of the large range that much copolymer mixture with which average weight ratios differ exists. The distributed holdout especially covering long duration becomes stability. Consequently, it becomes the cement dispersing agent which can respond also to fluctuation of W/C, or fluctuation of temperature enough.

[0027] the copolymer mixture (A-1) which the cement dispersing agent of this invention made the above-mentioned monomer (A1) react preferably by the mole ratio of the range of /(A1) (A2) =0.02-4, and was obtained as above-mentioned, although copolymer mixture (A-2) is contained further preferably (A2) Also in any, these mole-ratio (A1)/(A2) is changing in the middle of the reaction once [at least]. Any of an increment, reduction, and those combination are easy to be sufficient as change of this mole ratio. In this mole ratio, when [gradual] it is, and carrying out and making it change intermittently, especially the count of change has 1 - 5 desirable times 1 to 10 times. Moreover, although any of a linear change, an exponential change, and other change are sufficient when changing this mole ratio continuously, as for the degree of change, 0.2, further 0.0005 to 0.1, especially 0.001-0.05 are desirable from per [0.0001] minute. Moreover, this mole ratio has the desirable thing of mole-ratio (A1)/before and behind change (A2) which it is in the range of 0.02-4 at least any they are, and it is desirable that especially both mole-ratio (A1)/before and behind change (A2) is in the range of 0.02-4. Moreover, although various modes have change of a mole ratio as described above, it is desirable at least 0.05 and that the difference of the maximum of this mole-ratio (A1)/(A2) and the minimum value in all copolymerization reactions is especially in the range of 0.05-2.5 in any case.

[0028] Although the copolymer mixture (A) of this invention is obtained by the manufacture approach of having the process which (A1) / (A2) mole ratio is changed once [at least], and carries out a polymerization Dropping of a monomer (A2) is started to dropping initiation and coincidence of the



[0029] In addition, the thing of the AUW of a monomer for which a dropping flow rate is changed and is manufactured especially as mentioned above 50 to 100% 30% or more is desirable.

[0030] A polymerization reaction may be performed under existence of a solvent. As a solvent, ketones, such as ester; acetones [, such as aliphatic hydrocarbon; ethyl acetate,], such as alicyclic hydrocarbon; nhexanes [, such as an aromatic hydrocarbon; cyclohexane], such as lower alcohol; benzene, such as water, a methanol, ethanol, isopropanol, and a butanol, toluene, and a xylene, and a methyl ethyl ketone, can be mentioned. Also in these, handling is easy and water and lower alcohol are desirable from the soluble point of a monomer and a polymer.

[0031] A polymerization initiator can be added in a copolymerization reaction. As a polymerization initiator, organic peroxide, an inorganic peroxide, a nitril system compound, an azo system compound, a diazo compound, a sulfinic-acid system compound, etc. can be mentioned. 0.05-50-mol% of the addition of a polymerization initiator is desirable to the sum total of a monomer (A1), a monomer (A2), and other monomers.

[0032] A chain transfer agent can be added in a copolymerization reaction. As a chain transfer agent, a low-grade alkyl mercaptan and low-grade mercapto fatty acid, a thio glycerol, thiomalic acid, 2-mercaptoethanol, etc. can be mentioned. The reaction temperature of a copolymerization reaction has desirable 0-120 degrees C.

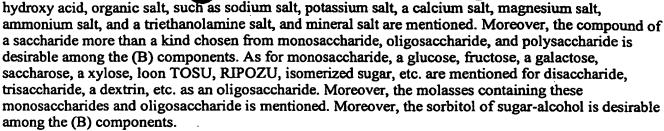
[0033] The obtained copolymer mixture can carry out deordorization processing if needed. Since an unpleasant smell tends to remain in a polymer when thiols, such as mercaptoethanol, are used especially as a chain transfer agent, it is desirable to carry out deordorization processing.

[0034] Although it is the copolymer mixture obtained by the above-mentioned manufacture approach and an acid type thing can be applied as a dispersant for cement also with an acid type, it is desirable to make it the form of a salt by neutralization by alkali from a viewpoint which controls hydrolysis of the ester by acidity. As this alkali, the hydroxide of alkali metal or an alkaline earth metal, ammonia, monochrome, JI, a trialkyl (carbon numbers 2-8) amine, monochrome, JI, a thoria RUKANORU (carbon numbers 2-8) amine, etc. can be mentioned. (Meta) When using an acrylic-acid system polymer as a dispersant for cement, it is desirable a part or to carry out full neutralization. The copolymer mixture salt in this invention means a part or the thing of a salt which carried out full neutralization for this acid type copolymer mixture. In addition, the copolymer mixture obtained in part, using a salt as a monomer (A2) is also contained in acid type copolymer mixture.

[0035] In addition, in order that the weight average molecular weight [the gel-permeation-chromatography method, polyethylene-glycol conversion, column:G4000PWXL+G2500PWXL (TOSOH CORP. make), eluate:0.2M phosphate buffer solution / acetonitrile =7/3 (volume ratio)] of the copolymer mixture obtained by the above-mentioned manufacture approach may acquire dispersibility sufficient as a dispersant for cement, 10,000-200,000 are desirable and 20,000-especially 100,000 are desirable.

[0036] In addition, further the monomer that can copolymerize acrylonitrile, acrylamide (meta), styrene, acrylic-acid (meta) alkyl (thing of carbon numbers 1-12 which may have hydroxyl group) ester, a styrene sulfonic acid, etc. may be used together. Although these can be used among [all] a monomer by 50 or less % of the weight and 30 more% of the weight or less of the ratio, 0 % of the weight is desirable.

[0037] [(B) Component] More than a kind of hydroxy acid chosen from a gluconic acid, glucoheptonic acid, an arabonic acid, a malic acid, and a citric acid is desirable among the (B) components. As a salt of



[0038] (B) As a component, hydroxy acid or its salt, and a saccharide are desirable, and especially a saccharide is desirable. In the severe condition by which concrete temperature exceeds 30 degrees C, hydroxy acid, such as monosaccharides, such as saccharose and a glucose, and a gluconic acid, or the salt of those is desirable.

[0039] [Cement dispersing agent] In the cement dispersing agent of this invention, if the dispersibility of a cement constituent, segregation resistance, on-the-strength manifestation nature, versatility, etc. are discovered good, the above-mentioned copolymer mixture (B) component [(A / [it is also called) the (A) component below]] can be used together by the ratio of arbitration. however, as for the amount of the (B) component, it is desirable to consider and decide the on-the-strength manifestation nature by coagulation delay of a cement constituent, as for both concrete weight ratio, (A) component / (B) component = 100 / 1 - 100/50 are desirable, 100 / 3 - 100/40 are more desirable, and 100 / 3 - 100/30 are still more desirable.

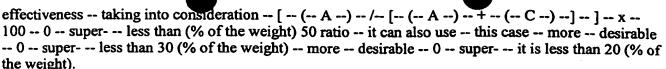
[0040] (A) To concrete, after mixing both beforehand, it may add, or you may add separately, and a component and the (B) component may be added after diluting with kneading water previously. [0041] The copolymer (C-1) obtained by copolymerizing the monomer (A1) and monomer (A2) which were shown in the cement dispersing agent of this invention of the aforementioned (A) component, without changing a mole ratio can be used together. What illustrated the monomer used for manufacture of a copolymer (C-1) by said monomer (A1) and monomer (B-2) is mentioned. By n in a general formula (a1), since engine performance differs, a copolymer (C-1) selects suitable n for demand characteristics in consideration of the class and loadings of the (A) component and the (B) component. n in (1) general formula (a1) is the copolymer (C-1-i) specifically using or more 1 less than 50 monomer. (2) The copolymer [n/in a general formula (a1)] using or more 50 less than 110 monomer (C-1-ii)

(3) The copolymer [n / in a general formula (a1)] using or more 110 300 or less monomer (C-1-iii) What is necessary is just to use together with the (A) component and the (B) component in consideration of **********, and each engine performance and application.

[0042] Moreover, the copolymer (C-2) which is made to carry out copolymerization of at least one sort (C'), and the maleic acid, its salt or anhydride (C") of the monomer expressed with the following general formula (c1) to the cement dispersing agent of this invention, and is obtained may be used together. As for the weight average molecular weight of a copolymer (C-2), 300-300,000 are desirable, and 5000-100,000 are more desirable. This molecular weight is measured like the (A) component. a copolymer (C-2) -- Maria Lim EKM, Maria Lim AKM (both Nippon Oil & Fats Co., Ltd.), and super one -- what is marketed by the trade name of 200 (electrochemistry company) can also be used.

Rc1O(Rc2O) rRc3 (c1) the inside of a formula, and the alkenyl radical of the Rc1:carbon numbers 2-5 -- desirable -- a vinyl group -- an allyl group and a metallyl radical -- more -- desirable -- the allyl group Rc2:carbon numbers 2-3 -- desirable -- the alkylene group Rc3:hydrogen atom of 2, or the alkyl group of carbon numbers 1-3 -- desirable -- methyl group r:2-150 -- desirable -- 2-90 -- more -- desirable -- 10-60 -- the number of 20-50 is expressed still more preferably.

[0043] The amount of these copolymers (C-1) used (it expresses with (C) hereafter (C-2)) (A) although what is necessary is to take the amount of a component and the (B) component into consideration, and just to determine suitably, when mainly concerned with the features of the (A) component [-- (C) / [(A) + (C) --] --] -- x100 -- 0 -- super- -- less than 50 (% of the weight) -- more -- desirable -- 0 -- super- -- it is less than 20 (% of the weight). moreover -- a copolymer -- mixture -- (-- A --) -- a copolymer -- (-- C --) -- mutual --



[0044] Moreover, to the cement dispersing agent of this invention, a melamine sulfonic-acid formalin condensate (C-3) may be used together. It is the well-known dispersant which the water-soluble salt of a melamine sulfonic-acid formalin condensate makes a bisulfite react to N-methylol-ized melamine which formaldehyde was made to react to a melamine and was obtained, carries out sulfo methylation of a part of methylol radical, subsequently adds an acid, makes carry out dehydration condensation of the methylol radical, makes it a formalin condensate, and is neutralized and obtained with alkali (for example, refer to JP,63-37058,B). As alkali, the hydroxide of alkali metal or an alkaline earth metal, ammonia, monochrome, JI, a trialkyl (carbon numbers 2-8) amine, monochrome, JI, a thoria RUKANORU (carbon numbers 2-8) amine, etc. can be mentioned. As a commercial item, there are my tee 150V-2 (Kao Corp. make), SMF-PG (Nissan Chemical Industries, Ltd.), a mel flow (Mitsui Chemicals, Inc.), mel MENTO F-10 (Showa Denko K.K. make), etc. As for the molecular weight of (C-3), 1000-100,000 and also 5000-50,000, especially 5000-20,000 are desirable (the gel-permeation-chromatography method, polystyrene sulfonate conversion). Moreover, as for the weight ratio of (C-3) and the above-mentioned copolymer mixture (A), (A)/(C-3) =100/1 - 100/100 are desirable, 100/5 - 100/50 are more desirable, and 100/5 - 100/30 are more desirable.

[0045] Moreover, water soluble polymers other than the above (C-4) which are rich in thickening nature can also be used together. As a water soluble polymer, a kind is [choose / out of following (C-4-1) - (C-4-8)] desirable in it being few.

(C-4-1) Nonionic cellulose ether, an acrylic-acid (C-4-2) copolymer, A polyalkylene glycol, the polysaccharide obtained by fermentation (C-4-4), (C-4-3) (C-4-5) To the alkylphenol which has xanthan gum, the monohydric alcohol of carbon numbers (C-4-6) 6-30, the monovalence mercaptan of carbon numbers 6-30, or the alkyl of carbon numbers 6-30, the amine of carbon numbers 6-30, or the carboxylic acid of carbon numbers 6-30 The alkylene oxide derivative which added an average of 10-1000 mols of alkylene oxide, (C-4-7) To the alkylphenol which has the monohydric alcohol of carbon numbers 6-30, the monovalence mercaptan of carbon numbers 6-30, or the alkyl of carbon numbers 6-30 The alkylene oxide derivative which added an average of 10-1000 mols of alkylene oxide, Some of a resultant with the compound which has one or more epoxy groups, polysaccharide (C-4-8), its alkylation, hydroxyalkyl-ized derivatives, or the hydrogen atom of all hydroxyl groups The hydrophobic substituent which has the hydrocarbon chain of carbon numbers 8-40 as a substructure (P), The polysaccharide derivative which it comes to permute by the ionicity hydrophilic radical (Q) which has as a substructure a radical more than a kind chosen from the group which becomes a sulfonic group, a carboxyl group, a phosphoric-acid radical, and a sulfate radical list from these salts.

[0046] The weight ratio of a water soluble polymer (C-4) and the (A) component When using the above (C-4-1), (C-4-2), (C-4-4), (C-4-6), and (C-4-7), as for both weight ratio, (A)/(C-4) = 100/1000 are desirable, and 100/0.2 - 100/1000 are more desirable. Moreover, when using the above (C-4-3), as for both weight ratio, (A)/(C-4) = 100/2.2 - 100/200 are desirable, and 100/2.2 - 100/3000 are more desirable. Moreover, when using the above (C-4-5) and (C-4-8), as for both weight ratio, (A)/(C-4) = 100/0.2 - 100/100 are desirable, 100/0.2 - 100/20 are more desirable, and especially 100/0.2 - 100/100 are desirable.

[0047] The cement dispersing agent of this invention can also contain other additives (**). For example, rosin soap, saturation or unsaturated fatty acid, a hydroxy sodium stearate, Lauryl sulfate, alkylbenzene sulfonic acid (salt), alkane sulfonate, The polyoxyalkylene alkyl (phenyl) ether, polyoxyalkylene alkyl (phenyl) ether phosphoric ester (salt), AE agents, such as a protein ingredient, an alkenyl succinic acid, and alpha olefin sulfonate; ** magnesium fluoride, Inorganic system retarders, such as a phosphoric acid or its salt, and the ester of boric acid; An amino carboxylic acid or its salt, Alkali meltable protein, a humic acid, a tannic acid, a phenol, polyhydric alcohol, organic system retarder [, such as phosphonic acid and its derivative,]; -- frothing

agent; -- thickener; -- silica sand; AE water-reducing agent; -- a calcium chloride -- Fusibility calcium salts, such as nitrous-acid calcium, a calcium nitrate, a calcium bromide, and a calcium iodide, Sulfates, such as chlorides, such as ferric chloride and a magnesium chloride, a potassium hydroxide, High-early-strength agents, such as a sodium hydroxide, a carbonate, a thiosulfate, formic acid (salt), and alkanolamine, or an accelerator; foaming agent; Resin acid (salt), waterproofing agent; blast-furnace-slag; plasticizer; defoaming agent; **** agents, such as fatty acid ester, fats and oils, silicone, paraffin, asphalt, and a wax, -- a; fly ash; high-range water reducing agent; silica fume; nitrite -- Rusr-proofers, such as phosphate and a zinc oxide; (meta) giant-molecule emulsions, such as acrylic-acid alkyl, are mentioned.

[0048] The cement dispersing agent of this invention is useful outside freshly mixed concrete and the concrete oscillating product field also in which field of the various concrete the object for self-leveling, the object for refractories, the object for plaster, the object for gypsum slurry, a light weight or the object for heavyweight concretes, the object for AE, the object for repair, the object for pre PAKKUDO, the object for tray me, the object for grouts, for the cold seasons, etc. As for especially the cement dispersing agent of this invention, it is desirable to be used by 0.05 - 2.0% of the weight of the ratio 0.01 to 5.0% of the weight to cement (as solid content).

[Example] The copolymer mixture shown in <copolymer mixture (A)> Table 1 and 2 was manufactured. A monomer (A1) and mole-ratio (A1)/(A2) of (A2) were changed in the middle of the reaction in that case.

[0050] [Table 1]

共 里合体 混合物	単量体	単量体	濟下時間	(A1)/(A2)	(A17/(A1")/(A2)	単量体混合物 流下分率			
	(A1") .	(A1")	(A2)	(分)	₩比 ⁴¹	重量比	海でガー	平均度量比 *2	
	MPEGMM(120)	MPEGMM (9)	MAA	O~55	0.36	55.0/28.8/16.2	61.1		
A-I	MPEGMM(120) MPEGMM (9)		MAA	55~75	0.59	55.0/33.7/11.3	22.2	13.9	
	MPEGMM(120)	MPEGMM (9)	MAA	75~90	0.83	55.0/36.3/8.7	16.7		
	MPEGMM(120)	MPEGMM (9)	MAA	0~55	0.37	60.0/25.6/14.4	61.1		
A-I	MPEGMM(120)	MPEQMM (9)	MAA	55~ 75	0.59	60.0/29.9/10.1	22.2	123	
	MPEGMM(120)	MPEGMM (9)	MAA	75~90	0.83	60.0/32.3/7.7	18.7		
А-Ш	MPEGMM(120)	MPEGMM (9)	MAA	0~45	1.13	55.0/38.4/6.6	50.0	7.3	
Α-Ш	MPEGMM(120)	MPEGMM (9)	·MAA	45~90	0.93	55.0/37.1/7.9	50.0	د. ا	

- *1:[(A1')+(A1")]/(A2)のモル比(以下同様)
- **★2:[(A2)の全量]/[(A1)の全量+(A1)の全量+(A2)の全量]の重量比(以下同様)**

[0051] [Table 2]

共選合体 混合物	単量体(A1) (A1")		単量体 (A2) 漢下時間 (分)		(A1)/(A2) 毛ル比	(A17)/(A177)/(A2) 重量比	単量体組合物 . 滴下分率 (重量%)	(A2) 平均重量比	
	MPEGMM(120)	MPEGMM (9)	MAA	0~55	0.36	55.0/28.8/16.2	61.1		
A-I-Na	MPEGMM(120) MPEGMM (8) MAA		MAA	55~75	0.59	55.0/33.7/11.3	22.2	13.9	
	MPEQMM(120)	MPEQMM (9)	MAA	75~90	0.83	55.0/36.3/8.7	16.7		
-	MPEQMM(120)	MPEQMM (8)	MAA	0~55	0.37	80.0/25.8/14.4	61.1	-	
A- II -Na	MPEQMM(120)	MPEQMM (9)	MAA	55~75	0.59	60.0/29.9/10.1	22.2	12.3	
	MPEQMM(120)	MPEQMM (9)	MAA	75~80	. 0.83	60.0/32.3/7.7	16.7		
A-III-Na	MPEGMM(120)	MPEQMM (9)	MAA	0.~45	1.13	55.0/38.4/6.6	50.0	7.0	
W-TT-MR	MPEGMM(120)	MPEGMM (9)	MAA	45~90	0.93	55.0/37.1/7.9	50.0	7.3	
A-IV-Na	MPEGMM (9)	-	MAA	0~15	0.43	71.2/-/28.8	16.7	22.0	
A-IV-Na	MPEGMM (8)		MAA	15~90	0.67	79.4/-/20.6	83.3	22.0	

[0052] (Note) MPEGMM is the abbreviation for methoxy polyethylene glycol monomethacrylate among Table 1 and 2, and the figure in () is the number of ethylene oxide average addition mols (the following -- the same). Moreover, MAA is a methacrylic acid (the following -- the same). Moreover, it means that "-Na" is sodium salt among Table 2. In addition, Table 1 and the copolymer mixture in two were manufactured according to the following examples of manufacture.

[0053] The water 329.9 weight section was taught to the <manufacture of example of manufacture 1:copolymer mixture A-I, and A-I-Na> glass reaction container, and the temperature up was carried out to 78 degrees C under nitrogen-gas-atmosphere mind after the nitrogen purge. Next, the 60% watersolution 216.4 weight section of MPEGMM (120), the 90% water-solution 75.5 weight section of MPEGMM (9), The mixed liquor of the methacrylic-acid 38.3 weight section, the 5% ammonium persulfate water-solution 27.7 weight section, and the 5%-2-mercaptoethanol water-solution 30.8 weight section are dropped in 55 minutes. The 60% water-solution 78.7 weight section of MPEGMM (120), the 90% water-solution 32.1 weight section of MPEGMM (9), The mixed liquor of the methacrylic-acid 9.7 weight section, the 5% ammonium persulfate water-solution 8.2 weight section, and the 5%-2mercaptoethanol water-solution 9.2 weight section are dropped in 20 minutes. Furthermore, the 60% water-solution 59.0 weight section of MPEGMM (120), the 90% water-solution 26.0 weight section of MPEGMM (9), The mixed liquor of the methacrylic-acid 5.6 weight section, the 5% ammonium persulfate water-solution 5.4 weight section, and the 5%-2-mercaptoethanol water-solution 6.0 weight section were dropped in 15 minutes. Mole-ratio (A1)/(A2) for every drop time is shown in Table 2. After dropping termination, after making it ripe at 78 degrees C for 60 minutes, the ammonium persulfate water-solution 20.7 weight section was added in 5 minutes 5%. It riped at 78 degrees C for 120 more minutes, the polymerization reaction was completed, and copolymer mixture A-I shown in Table 1 was obtained.

[0054] Furthermore, the sodium-hydroxide water-solution 20.8 weight section was added 48%, it neutralized, and copolymer mixture A-I-Na shown in Table 2 was obtained. pH (20 degrees C) of the 5-% of the weight water solution of this copolymer mixture A-I-Na was 5.9.

[0055] In addition, in Table 1 and 2, copolymer mixture A-II and A-II-Na to which the mole ratio of (A1)/(A2) was changed twice were manufactured according to this example 1 of manufacture. [0056] The water 423 weight section was taught to a <manufacture of example of manufacture 2:copolymer mixture A-IV-Na> thermometer, an agitator, a dropping funnel, nitrogen installation tubing, and the glass reaction container equipped with the reflux condenser, and the nitrogen purge was

performed. After carrying out a temperature up to 70 degrees C under nitrogen-gas-atmosphere mind. MPEGMM (9) Then, the 44.9 weight sections, 3 liquid of the monomer liquid for dropping (1) and the 5%-2-mercaptoethanol acid water-solution 14.2 weight section which mixed the methacrylic-acid 18.2 weight section, and the 5% ammonium persulfate water-solution 13.8 weight section is dropped at coincidence in 15 minutes. Subsequently MPEGMM (9) The 250.5 weight sections, 3 liquid of the monomer liquid for dropping (2) and the 5%-2-mercaptoethanol acid water-solution 59.2 weight section which mixed the methacrylic-acid 65.2 weight section, and the 5% ammonium persulfate water-solution 57.6 weight section was dropped in 75 minutes, and dropping was terminated in a total of 90 minutes. After dropping termination, after riping by this ** for 1 hour and dropping the ammonium persulfate water-solution 21.4 weight section over 10 minutes 5%, it was made to ripe at 70 degrees C for 2 hours, and the polymerization reaction was completed. The sodium-hydroxide water-solution 57 weight section was added 48 more%, it neutralized, and copolymer mixture A-IV-Na shown in Table 2 was obtained. pH of the 5-% of the weight water solution of this copolymer mixture A-IV-Na was 6.0 (20 degrees C). [0057] In addition, in Table 1 and 2, copolymer mixture A-III and A-III-Na to which the mole ratio of (A1)/(A2) was changed once were manufactured according to this example 2 of manufacture. [0058] The (B) component shown in <(B) Component> table 3 was used. [0059]

B-I	グルコン酸ナトリウム
B-I	グルコース
B-II	サッカロース
8-W	デキストリン (パインデックス3、松谷化学)
B-V	サカロースとクエン酸の等量(重量比)混合物
B-VI	リンゴ酸
B- M	ソルビトール

[0060] The copolymer shown in the <copolymer (C)> table 4 was manufactured. The monomer (C1) and the weight ratio of (C2) presupposed that it is fixed at that time. [0061]

[Table 4]

共置合体	単 量体 (C1)	単量体(C2)	(C2)宜量比 ⁴¹							
C- I	MPEGMM (9)	MAA	22.0							
C-1	мредмм (75)	MAA	9.2							
С-ш	MPEGMM (120)	MAA	12.8							
C-M	MPEGMM (120)	MAA	6.0							
C-A	AE	MA:	6.0							
с–и	マイティ150V-2 (メラミンスルホン酸ホルマリ)	/縮合物、花王(4	妹))							
C-AX	オクタテ"シル(世後度0.0032)-3-スルホー2-ヒト"ロキシフプロピル(世後度0.332)-とト"ロキシエチルセルロース									
C-AII	オレイルアルコールのエチレンオキサイト、平均200モル付加物									
c-ix	C-面とピニルシクロヘキセンシ゚エポキシドとの反応物									
C-X	ポリエチレングリコール(蛍Ქ	量平均分子量20	000)							

*1:(C2)/[(C1)+(C2)]の重量比

[0062] (Note) AE is the abbreviation for methoxy polyethylene-glycol (n= 40) monoallyl ether among Table 4, MA is a maleic anhydride and MAA is a methacrylic acid.

[0063] <Concrete test condition> (1) ingredient W= tap water C= ordinary portland cement (specific gravity = 3.16)

LC= limestone impalpable powder (specific gravity =2.70, specific surface area of cement by blaine = 5200)

Fine aggregate = from Kanto Kimitsu (specific gravity = 2.63)

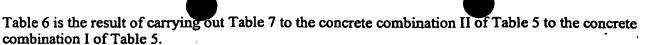
Coarse aggregate = osteoclasis from Ibaragi (specific gravity = 2.62)

W/C -- combination of the concrete prepared with the (2) combination above-mentioned ingredient = (unit weight of unit weight/C of W) x100%second/a=[fine aggregate volume/(fine aggregate volume + coarse aggregate volume)] x100% is shown in Table 5.
[0064]

[Table 5]

	W/(C+LS) (宜量%)	s/a (体積%)	W (kg∕m³)	C (kg/m³)	LS (kg/m³)
配合 I	42.5	48.0	170	400	0
配合Ⅱ	33.0	49.0	165	350	150

[0065] (3) The ingredient and dispersant for 30l. of performance-evaluation concrete were fed into the compulsive biaxial mixer (50l.), it kneaded for 90 seconds, and the following engine performance immediately after discharge was evaluated. Moreover, Combination I is 26 degrees C (concrete temperature of 28 degrees C) in room temperature, and carried out combination II at the room temperature of 31 degrees C (concrete temperature of 33 degrees C). In addition, the evaluation approach was changed as follows by Combination I and II. Although a result is shown in Tables 6 and 7,



[0066] (3-1) The rate of addition to the total fine particles of the dispersant solid content taken to set the initial slump index (JIS-A1101) of the rate of <evaluation by Combination I> (3-1-1) addition to 23**1cm is measured. Dispersibility is so good that a numeric value is small.

[0067] (3-1-2) The percentage of the slump index 60 minutes after receiving the initial slump index of holdout. Distributed holdout is so good that a numeric value is large.

[0068] (3-1-3) The mortar which separated and obtained coarse aggregate from the concrete after measuring a flowing-down time amount slump index by the screen of 5mm of openings To the equipment of the configuration of <u>drawing 1</u> which processed and produced stainless steel (SUS304) After being filled up where lower discharge opening is closed, and cutting by rubbing in respect of up injection opening, Lower discharge opening was opened wide, when gravity flow of the mortar was carried out and it was visually observed from up injection opening, time amount (flowing-down time amount) until a hole is checked by some mortar [at least] was measured, and this was used for viscous evaluation. The viscosity of concrete is so low that flowing-down time amount is short, and segregation resistance is weak.

[0069] (3-1-4) Conclusion time amount was measured by ASTM-C403 law to the mortar which measured setting-time flowing-down time amount.

[0070] (3-2) The rate of addition to the total fine particles of the dispersant solid content taken to be set to rate slump flow-value [hi-performance concrete construction guide (concrete library 93)] of <evaluation by Combination II> (3-2-1) addition 650**10mm is measured. Dispersibility is so good that a numeric value is small.

[0071] (3-2-2) The percentage of the slump index 60 minutes after receiving the initial slump flow value of holdout. Distributed holdout is so good that a numeric value is large.

[0072] (3-2-3) The mortar which separated and obtained coarse aggregate from the concrete of 650**10mm of flowing-down time amount slump flow values by the screen of 5mm of openings To the equipment of the configuration of drawing 1 which processed and produced stainless steel (SUS304) After being filled up where lower discharge opening is closed, and cutting by rubbing in respect of up injection opening, Lower discharge opening was opened wide, when gravity flow of the mortar was carried out and it was visually observed from up injection opening, time amount (flowing-down time amount) until a hole is checked by some mortar [at least] was measured, and this was used for viscous evaluation. The viscosity of concrete is so low that flowing-down time amount is short, and segregation resistance is weak.

[0073]

[Table 6]

_				- 44.4.1		-							
				混合物の)		(B) A		共宣合体(C)		産加率	保持性	遊下時間	硬化時間
		種類	重量%	種類	盘量%	種類	重量%	種類	重量%	(96)	(%)	(秒)	(時一分)
Ħ	1-1	A-11 O(≠12,3)	100	1	-	-	_	ł	_	0.23	81	17.5	5-30
報例	1-2	•	-	-	1	8-11	7.5	O- I (Y=22.0)	92.5	0.27	75	15.9	9-00
1971	1-3	-	-	_	1		7.5	C-17 (Y=8.0)	92.5	0.24	72	20.0	6-05
	1-1	A-11 (X=12.3)	92.5	. –	- .	B-11	7.5	-	•	0.25	80	18.5	8-15
	1-2	A-II (X=12.3)	91.0	_	_	₽-7	8.0	_	-	0.25	78	17.1	6-20
	1-3	A-11 O(=12.3)	89.5	-	-	8-I	10.5	-	1	0.25	78	18.3	6-15
	1-4	A-II (X=123)	88.0	-	_	B- I	12.0	-	-	0.26	78	18.5	6-10
	1-6	A-II (X=123)	88.5	-		B-AX	12.5	. –	-	0.27	75	18.8	6-20
	1-6	A-II (X=12.3)	85.0	_	-	B-I7	15.0	_	-	0.27	75	17.8	6- 15
実	1-7	A-II (X=12.3)	80.0	_	_	B-IV	25.0	_	-	0.27	. 81	18.7	7-05
施	1-8	A-II (X=12.3)	65.0	-		B- IV	35.0	_	-	0.30	86	19.3	7–40
例	1-0	A-II (X=12.3)	57.5	A-Ⅲ (X=7.2)	35.0	B-II	7.5	_	1 0-0	0.27	85	18.6	6-05
	1-10	A-11 (X=12.3)	45.0	A-III CX=7.8)	45.0	B-II	10.0	_	_	0.28	80	18.9	8 −1 5
	1-11	A-II-Na (X=12.3)	92.5	-	-	B-II	7.5	_	-	0.26	83	18.4	6 –15
	1-12	A-II-Na (X=12.3)	89.5	_	-	B-I	10.5	. -	-	0.25	77	18.4	0 –15
	1-13	A-II-Na (X=12.3)	88.0	_	-	B∽I	12.0	_	-	0.26	78	18.7	8-10
	1-14	A- I -Na CX=13.9)	57.5	A-III-Na (X=7.3)	35.0	B-1X	7.5	_	-	0.27	84	18.9	6 –05
	1-15	A- I -Na (X=13.8)	85.0	A-IV-Na (X=22.0)	25.0	B-11	10.0	-	-	0.29	80	17.0	8-45

[0074] the average weight ratio [as opposed to all the monomers in the case of manufacture of copolymer mixture (A) in front Naka and X] of a monomer (A2) and Y are the average weight ratios of a monomer (C2) to all the monomers in the case of manufacture of a copolymer (C) (the following -- the same).

[0075] The example 1-1 of a comparison shows that holdout is not enough in the case of 28-degree C concrete temperature in (A) component independent. Moreover, although holdout will improve rather than the example 1-1 of a comparison if the (B) component is used together from the example 1-2 of a comparison to a copolymer with the small number of EO addition mols (C), it turns out that the setting time falls. Moreover, although holdout will improve rather than the example 1-1 of a comparison if the (B) component is used together from the example 1-3 of a comparison to a copolymer with the large number of EO addition mols (C), increase of the viscosity of concrete is large and it turns out for a freshly-mixed-concrete application that there is a possibility that pump pumpability may fall. On the other hand, it turns out that delay of the setting time is controlled to the minimum, without improving holdout and increasing the viscosity of concrete from -15, if the (B) component is used together for an example 1-1 - 1(A) component. When that from which the average weight ratio X differs as an example 1-9, 1-10, 1-14, and a 1(A) component is especially used together from -15, it turns out that holdout is improvable, maintaining other engine performance to stability.

[0076]

[Table 7]

Г		#	(混合物(A)		(B)	议分		共宜色	体(C)		承加率	保持性	競下時間
L		種類	筻量%	種類	量量%	種類	堂量%	種類	選量%	種類	重量96	(%)	(%)	(秒)
比較例	2-1	A- I (X=13.9)	60	A-III (X=7.3)	. 35	1	ı	C-II (Y=12.6)	5	-	-	0.23	50	12.5
	2-1	A- I (X=13.9)	50	A-Ⅲ (X=7.3)	30	B-II	15	C-Ⅲ (Y=12.6)	5	-	-	0.28	80	13.8
	2-2	A- I (X=13.9)	47	А-Ш (X=7.3)	21	8-11	16	C-II (Y=12.6)	5	C- I (Y=22.0)	11	0.26	78	12.8
	2-3	A- [(X=13.9)	47	A-Ⅲ (X=7.3)	21	田田	16	C-II (Y=12.6)	5	C-II (Y=9.2)	11	0.24	75	13.1
	2-4	A- [(X=13.9)	49	A-III (X=7.3)	27	ㅂ	· 12	O-II (Y=12.6)	4	C-▼ (Y=6.0)	8	0.28	84	13.1
実施例	2-5	A- I (X=13.9)	49	А-Ш ·(X=7.3)	27	B-II	12	C-II (Y=12.6)	4	C-VI	8	0.29	88	14.3
例	2-6	A~ I (X=13.9)	54	A-Ⅲ (X=7.3)	29	B-II	13	C≻Ⅲ (Y=126)	3.99	C-AI	0.01	0.28	80	17.0
	2-7	A-I (X=13.9)	54	A-III (X=7.9)	29	B-111	12	C-Ⅲ (Y=12.6)	4.2	C-A	0.8	0.26	81	16.1
	2–8	A- [(X=13.9)	53.9	A-II (X=7.3)	29	B-II	12.4	C-II (Y=126)	4.1	C-IX	8.0	0.28	80	15.3
	2-9	A- I (X=13.9)	52	A-II (X=7.3)	28	B-II	12	C-II (Y=12.6)	4	с-ж	4	0.26	81	14.9
	2-10	A-I-Na (X=13.9)	54	А-Ш-Na (X=7.3)	29	8-II	13	C-II (Y=126)	3.99	C-AII	0.01	0.26	82	15.3

[0077] When concrete temperature becomes 33 degrees C from the example 2-1 of a comparison by the concomitant use system of a copolymer (C) with large (A) component and number of EO addition mols, it turns out that the fall of holdout is remarkable. On the other hand, when the (B) component is used together in the system of the example 2-1 of a comparison from an example 2-1 to 2-6, it turns out that holdout is improvable. It turns out that the improvement effect of the holdout which was excellent in it especially even if it used together the still better known dispersant and water soluble polymer in the system of an example 2-2 to 2-9 to the example 2-1 is acquired. Moreover, it turns out that a result good also when the sodium salt of copolymer mixture is used is obtained from an example 2-10.

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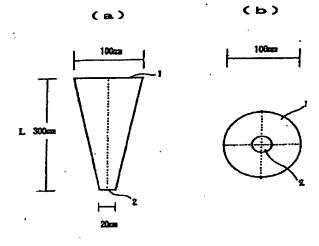
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DRAWINGS

[Drawing 1]



[Translation done.]

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